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IS 10115 (1982): Phosphorus pentasulphide [CHD 1: Inorganic Chemicals]



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IS : 10115 - 1982

Indian Standard
**SPECIFICATION FOR
PHOSPHORUS PENTASULPHIDE**

UDC 546 185 221



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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 4

December 1982

AMENDMENT NO. 2 JUNE 2005
TO
IS 10115 : 1982 SPECIFICATION FOR PHOSPHORUS
PENTASULPHIDE

(Page 5, Appendix A, clause A-2.0, lines 2 and 3) — Substitute ' PO_4^{-3} ', for ' PO_4-3 '

(Page 6, Appendix A, clause A-2.1.5, line 2) — Substitute ' MoO_3 ' for ' MoO_3 '

(Page 11, Appendix A, clause A-5.5, line 3) — Insert '14' between '10' and '20'

(CHD 1)

Reprography Unit BIS New Delhi India



AMENDMENT NO. 1 APRIL 1984

TO

IS:10115-1982 SPECIFICATION FOR PHOSPHORUS
PENTASULPHIDE

Corrigendum

(Page 7, clause A-2.3) - Substitute '4.768'
for '01 735'.

(CDC 3)

Reprography Unit, ISI, New Delhi, India

Indian Standard

SPECIFICATION FOR PHOSPHOROUS PENTASULPHIDE

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Indian Standard
SPECIFICATION FOR
PHOSPHOROUS PENTASULPHIDE

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 24 February 1982, after the draft finalized by the Inorganic Chemicals (Misc) Sectional Committee had been approved by the Chemical Division Council.

0.2 Phosphorous pentasulphide (P_2S_5) is used as an intermediate in pesticides and oil additives.

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS . 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for phosphorous pentasulphide

2. GRADES

2.1 The material shall be of the following two grades:

- a) Grade A — for use in oil additives, and
- b) Grade B — for use in pesticides.

3. REQUIREMENTS

3.1 Description — The material shall be in the form of a fine, free-flowing powder, yellow to pale greenish in colour and free from extraneous impurities.

3.2 Particle Size — The particle size of the material shall be as agreed to between the purchaser and the supplier.

*Rules for rounding off numerical values (*revised*)

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3.3 The material when tested as prescribed in Appendix A, shall also comply with the requirements given in Table 1. Reference to relevant clauses of Appendix A is given in col 4 of the table.

TABLE 1 REQUIREMENTS FOR PHOSPHOROUS PENTASULPHIDE

Sl No (1)	CHARACTERISTIC (2)	REQUIREMENT (3)		METHOD OF TEST (Ref to Cl No in Appendix A) (4)
		Grade A	Grade B	
i)	Phosphorus content (as P)	27.2 to 27.7	27.8 to 28.4	A-2*
ii)	Sulphur content (as S)	72.2 to 72.8	71.6 to 72.2	A-3
iii)	Melting point	286 - 290°C	—	A-4
iv)	Iron (as Fe), ppm, Max	100	—	A-5
v)	Reactivity, °C/minute, Min	0.8	—	A-6

*If desired by the purchaser, any other standard method may be used as agreed to between the purchaser and the supplier.

4. PACKING AND MARKING

4.1 Packing — The material shall be packed in mild steel drums lined with polyethylene film or as agreed to between the purchaser and the supplier.

4.1.1 For Grade A of the material, the container shall also be inerted, using dry nitrogen/carbon dioxide or other inert gas to protect the material from other reaction

4.2 The containers shall be securely closed and shall bear legibly and indelibly the following information and other labelling provisions applicable to the material.

- Name of the material;
- Name of the manufacturer and his recognized trade-mark, if any;
- Gross and net mass,
- Date of manufacture,
- Batch number, and
- Highly toxic by inhalation, strong irritant, dangerous fire risk, ignite by friction. Contact with water or acids liberates hydrogen sulphide gas.

4.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

5. SAMPLING

5.1 The method of drawing representative samples of the material from a lot, and the criteria for conformity shall be as prescribed in Appendix B.

APPENDIX A

(Clause 3.3)

METHODS OF TEST

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis

A-2. PHOSPHOROUS CONTENT

A-2.0 Outline of the Method — Phosphorous in phosphorous pentasulphide is oxidized into PO_4-3 by oxidation using sulphuric acid invert aqua regia and bromine. The PO_4-3 ions are precipitated as quinoline phosphomolybdate, filtered, dissolved in excess of standard alkali and back titrated with standard acid using phenolphthalein as indicator and phosphorus is calculated volumetrically.

*Specification for water for general laboratory use (*second revision*).

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A-2.1 Reagents

A-2.1.1 Concentrated Nitric Acid — see IS : 264-1976*.

A-2.1.2 Concentrated Hydrochloric Acid — see IS : 265-1976†.

A-2.1.3 Bromine

A-2.1.4 Dilute Sulphuric Acid — Relative density 1.21. Slowly add 200 ml of cooled concentrated sulphuric acid to about 700 ml of water contained in a 1 000 ml volumetric flask. Stir and make up to 1 000 ml with water.

A-2.1.5 Citric Molybdate Solution — Weigh 50 g of molybdate anhydride (MoO_3) in a 500-ml beaker. Add 200 ml of water and then 11 g of sodium hydroxide pellets. Heat the beaker until molybdic anhydride dissolves. Dissolve 60 g of citric acid crystals in 250 ml of water in a one litre beaker and add 140 ml of hydrochloric acid. Pour the molybdic solution into the citric acid solution, stirring vigorously all the time. Cool the solution and filter it into a one litre volumetric flask. Make up to one litre with water. The solution, may be green or blue, depending on exposure to light. If necessary add a dilute solution of potassium bromate (0.5 to 1 percent) dropwise until green colour disappears. Keep solution in a dark place in a well stoppered bottle, made of polyethylene.

A-2.1.6 Quinoline Solution — Dilute 60 ml of concentrated hydrochloric acid in a beaker with 300 to 400 ml of water and heat to 70° to 80°C. Slowly add 25 ml of quinoline solution, stirring all the while. The quinoline dissolves, cool the solution, dilute it with water to one litre and filter. Keep solution in a polyethylene bottle.

A-2.1.7 Standard Sodium Hydroxide — 0.1 N

A-2.1.8 Standard Hydrochloric Acid — 0.1 N.

A-2.1.9 Mixed Indicator Solution — Two volumes of 0.1 percent of phenolphthalein in 60 percent rectified spirit, three volumes of 0.1 percent thymol blue (0.1 g indicator, 22 ml of 0.1 N NaOH and diluted to 100 ml, 50 ml methylated spirit diluted to 100 ml.

A-2.2 Procedure

A-2.2.1 Grind and weigh accurately about one g of the material. Transfer to a 500-ml flask with a ground glass neck. Fit reflux condenser to it and add 20 ml of concentrated hydrochloric acid followed by 60 ml concentrated nitric acid via the condenser. Allow reaction to take place under a fume

*Specification for nitric acid (second revision)

†Specification for hydrochloric acid (second revision)

cupboard for about 30 minutes, and then heat on a steam bath. Add 5 ml of bromine, and reheat in steam bath until bromine disappears. Cool and rinse condenser with water. Cover the flask with small funnel and boil on a low flame till a final volume of few ml is attained. Add water and transfer solution totally into the 1 000 ml volumetric flask. Make up to one litre and filter off any insoluble residue through a dry fluted filter into a dry 1 000 ml flask, discarding the first portion of the filtered liquid

A-2.2.2 Using volumetric pipette, draw off 25 ml of the solution and pour it into a 500-ml flask. Add 10 ml of dilute sulphuric acid and boil for 5 minutes. Dilute the solution with water to 150 ml and add 25 ml of citric molybdate solution, heat to boiling and add quinoline solution dropwise from a burette until about one ml has been added. Boil again and to the gently boiling solution add the reagent, a few ml at a time with stirring until 12.5 ml in all have been introduced. A coarsely crystalline precipitate is thus obtained which can easily be filtered.

A-2.2.3 Filter under suction through a thick pad of filter pulp prepared on a perforated disc in a funnel. Wash the precipitate and the flask with water till free from acid (About 10 ml of the washing and 4 drops of the indicator should show colour change from violet to yellow with 1 drop of 0.1 N sodium hydroxide solution). Transfer the pad of filter paper back with the precipitate to the original flask. Add 50 to 60 ml of water and then add 0.1 N sodium hydroxide solution with stirring in multiples of 25 ml from a pipette till the precipitate dissolves completely. Add 5 to 6 drops of the indicator solution and titrate the excess of alkali with standard hydrochloric acid till violet colour changes to yellow. The end point is sharp.

A-2.2.4 Run a blank determination on the same quantity of sodium hydroxide solution which was added in the test and titrate with standard hydrochloric acid using 5 to 6 drops of indicator solution.

A-2.3 Calculation

$$\text{Phosphorous (as P) percent by mass} = \frac{(V_1 - V_2) \cdot N \times 0.1735}{M}$$

where

V_1 = volume in ml of standard hydrochloric acid required for the blank,

V_2 = volume in ml of standard hydrochloric acid required for the sample,

N = normality of standard hydrochloric acid, and

M = mass in g of the material taken for the test.

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A-3. SULPHUR CONTENT

A-3.0 Outline of the Method — Phosphorous pentasulphide is first dissolved in sodium hydroxide and then oxidized first with hydrogen peroxide and then further with bromine to phosphoric acid and sulphuric acid. The sulphate ions are precipitated as barium sulphate and estimated as sulphur.

A-3.1 Reagents

A-3.1.1 Barium Chloride Solution ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$) — 10 percent (m/v).

A-3.1.2 Hydrogen Peroxide — 30 percent (v/v).

A-3.1.3 Sodium Hydroxide Solution — 10 percent (m/v).

A-3.1.4 Silver Nitrate Solution — 0.1 N.

A-3.1.5 Bromine

A-3.1.6 Hydrochloric Acid

A-3.2 Procedure

A-3.2.1 Weigh accurately about 1.0 g of the material and transfer it to a flask containing 100 ml of sodium hydroxide solution. Warm gently to about 50°C and stir until the material is completely dissolved. Cool the solution and then add 20 ml of hydrogen peroxide cautiously in small increments. Add one to two boiling chips or glass beads and carefully boil the solution gently for 10 minutes. Cool and add another 20 ml of hydrogen peroxide cautiously in small increments. Again boil the solution gently for 10 to 20 minutes.

A-3.2.2 Dilute the solution to about 100 ml with water and cool. Add 5 ml of bromine, cool and add hydrochloric acid dropwise (*see Note*) until no bromine evolves; then add about 3 ml in excess. Boil until the evolution of bromine ceases. Cool the sample to room temperature and transfer quantitatively to a 250-ml volumetric flask, avoiding the transfer of the boiling chips. Dilute to the mark with water and mix. Pipette out 10 ml of the this solution into a 400-ml tall beaker. Dilute to about 200 ml. Heat the solution to boiling, add dropwise from a burette 10 ml of barium chloride solution, at the rate of about 1 ml/min. Stir the solution constantly during the addition. Allow the precipitate to settle for a minute or two, then test the supernatant liquid for complete precipitation by adding a few drops of barium chloride solution. If a precipitate is formed, add more barium chloride solution, repeat this operation again, until an excess of barium chloride is present.

A-3.2.3 Cover the solution and keep it hot, on a water bath, in order to allow time for complete precipitation. Test the clear supernatant liquid with a few drops of barium chloride solution for complete precipitation. Filter through ashless filter paper (Whatman 42 or equivalent) and wash the precipitate with small portions of hot water jet. Continue the washing until the washings are free from chloride ion (5 ml of the wash solution gives no opalescence with a drop or two of silver nitrate solution). Eight or ten washings are usually required.

A-3.2.4 Place the paper and precipitate in a porcelain/silica crucible, previously ignited to redness, cooled in a desiccator and weighed. Dry the paper by placing the loosely covered crucible upon a triangle several centimetres above a small flame. Gradually char the paper by increasing heat and finally ignite in a muffle furnace at 775 to 800°C for ten minutes. Cool in a desiccator and weigh.

NOTE — Bromine fumes are released when this solution is boiled. This addition of hydrochloric acid and boiling must be done in fuming cupboard as bromine fumes are very toxic.

A-3.3 Calculation

$$\text{Sulphur, percent by mass} = \frac{M_1 \times 343.4}{M}$$

where

M_1 = mass in g of the residue, and

M = mass in g of the material taken for the test.

A-4. DETERMINATION OF MELTING POINT

A-4.1 Apparatus — Fisher-John's melting point apparatus Cover glasses for microscope slides, 18 mm in diameter

A-4.2 Procedure — A very small quantity of the finely pulverized material is placed between two cover glasses. The cover glasses are placed on the heating stage of the apparatus. The heating current is turned on and the sample is observed in the magnifier. The melting point is observed when small droplets appear between the cover glasses. The temperature can be raised rapidly to within 10 of the melting point, but it should be raised slowly after that.

A-4.3 A second cover glass is unnecessary when the melting point of fats, greases and waxes is determined. Because of the uncertainty of there melting points a tiny spike is formed on the cover glass and the melting point is obtained when the tip bends or begins to flow.

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A-5. DETERMINATION OF IRON

A-5.1 Apparatus

A-5.1.1 Electrophotometer, equipped with a blue filter at $440^\circ \text{ m}\mu$ or a spectrophotometer.

A-5.1.2 *Graduated Cylinders* — 100 ml.

A-5.1.3 *Filter Paper* — Munktel's No. 00 or equivalent.

A-5.1.4 *Porcelain Crucible*

A-5.1.5 *Muffle Furnace*

A-5.1.6 *Volumetric Flask* — 100 ml capacity

A-5.1.7 *Pipette* — 10 ml capacity.

A-5.1.8 *Burette* — 50 ml capacity.

A-5.2 Reagents

A-5.2.1 *Ammonium Thiocyanate*

A-5.2.2 *Hydrochloric Acid* — see IS : 265-1976*.

A-5.2.3 *Potassium Persulphate* — solid.

A-5.2.4 *Nitric Acid* — see IS : 264-1976†.

A-5.2.5 *Potassium Bisulphate or Pyrosulphate* — solid

A-5.2.6 *Standard Iron Wire*

A-5.2.7 *Ammonium Thiocyanate Solution* — 1 N

A-5.2.8 *Dilute Hydrochloric Acid* — 1 : 1 (v/v).

A-5.3 Procedure — A sample containing 0.05 to 0.1 mg iron is placed in a 250 ml beaker to which has been added about 50 ml water, 5 ml of HCl and 0.1 g of potassium persulphate. Solution may be accomplished with heat but the resultant solution must be colourless. For organic sample, the material is weighed into a porcelain crucible, ignited and fired in a muffle furnace until no carbon remains. The ash is treated with about one gram of potassium bisulphate and heated over a burner until the ash is dissolved. The crucible is cooled and placed in a 250 ml beaker containing 50 ml of water and 5 ml of hydrochloric acid is heated to dissolve the melt.

*Specification for hydrochloric acid (second revision)

†Specification for nitric acid (second revision)

A-5.3.1 The contents of the beaker are brought to a boil and filtered through Munktell's No. 00 paper into a 100 ml graduated cylinder. The paper is washed twice with hot water but care must be taken to avoid a total of more than 80 ml in the graduate. The solution is cooled and treated with 10 ml of 1 N ammonium thiocyanate solution. The volume is made up to 100 ml with water and mixed thoroughly by transferring the solution to a 250 ml beaker.

A-5.3.2 The transmission of light through the solution is measured in percent by use of the electrophotometer equipped with a blue filter (440 m μ) or a spectrophotometer with the wave length set at 440 m μ . The transmission of light through distilled water is considered 100 percent transmission. The amount of iron present in mg is taken from a curve prepared in the manner described below.

A-5.4 Calculation

$$\text{Iron (as Fe), percent by mass} = \frac{\text{mg Fe (obtained from curve)}}{\text{Sample mass taken for test} \times 10}$$

A-5.5 Preparation of Standard Calibration Curve— Into eight 250 ml beakers are placed varying amounts of the iron solution (0.010 mg Fe per ml) i.e. 1, 2, 4, 6, 8, 10, 20 ml corresponding respectively to 0.01, 0.02, 0.04, 0.06, 0.08, 0.10, 0.14 and 0.20 mg Fe. To each of these solutions is added about 25-30 ml water, 10 ml, 1 : 1 hydrochloric acid, 10 ml 1N NH_4CNS and about 0.1 g potassium persulphate. The solutions are mixed well and diluted to 100 ml by using a 100 ml graduated cylinder. The transmission of light of each solution is measured with 100 percent transmission assumed on pure water. A curve is plotted, the abscissa representing concentration in mg, the ordinate representing the percent transmission of light.

NOTE 1 — The orange colour of the iron thiocyanate complex is unstable and the light transmission must be measured within ten minutes after the development of the colour.

NOTE 2 — When the colour is developed in the 100 ml graduated cylinder the total acidity should be equal to 5 ml of HCl or 10 ml of 1 : 1 HCl. If large amounts of phosphate or other buffering agents are present, the acidity must be increased accordingly, but the results will be nevertheless suspect.

NOTE 3 — In samples containing barium and/or lead, the sulphate and persulphate ion should be avoided, with a few drops of nitric acid being used as an oxidizing agent.

NOTE 4 — Since potassium bisulphate contains an appreciable amount of iron, the iron should be determined on a blank containing all the chemicals used in the determination. This blank should be subtracted from the total percent iron.

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A-6. REACTIVITY

A-6.1 Apparatus — Dewar flask, wide-mouth, one pint size (Fisher Scientific 10-195 is satisfactory).

A-6.1.1 Stirrer motor, variable speed, capable of operating at 120 rpm, equipped with a two-bladed glass stirrer having the following dimension. The two blades shall have a pitch of 45°. Each blade shall be 13 mm long and the diameter inscribed by the rotating blades shall be not more than 30 mm.

A-6.1.2 *Stopper for Dewar flask* — equipped with glass-lined holes for stirrer and thermometer, and vented for escape of gas.

A-6.1.3 Thermometer, standard laboratory, 650 mm, graduated to 101°C, in 0.1°C divisions.

A-6.1.4 *Mortar and pestle*

A-6.1.5 *IS Sieve* — 300 micron.

A-6.1.6 *Graph paper* — 25.4 × 25.4 per cm.

A-6.2 Reagent — Isopropyl alcohol, technical grade, 99 percent.

A-6.3 Procedure — The stirring rate of the motor is adjusted to 120 rpm. The stirrer and thermometer are inserted through the sleeves of the stopper and positioned 1.27 cm from the bottom of the vessel, making sure that the stirrer does not hit either the side of the flask or the thermometer. Using a graduate, 100 ml of isopropyl alcohol, adjusted to $30 \pm 1^\circ\text{C}$, is added to the reaction flask. Agitation is started and 36 ± 0.1 g of phosphorus pentasulphide, ground and passed through a 60 mesh Sieve, is added to the reaction flask. The reaction is allowed to proceed until temperature rise increments are constant. This is accomplished by plotting a graph, time in minutes versus temperature, recording the reaction temperature every four minutes for one hour.

The end-point is taken as either the maximum temperature reached by the reaction mixture or the point at which the rate of temperature change becomes constant, the point beyond which the curve becomes a straight line

A-6.4 Calculation

$$\text{Reactivity Rate, } ^\circ\text{C/minute} = \frac{\text{Maximum temperature} - \text{Initial temperature}}{\text{Time required for temperature rise}}$$

Reactivity Rate is reported to the nearest 0.1 °C/minute.

APPENDIX B(*Clause 5.1*)**SAMPLE OF PHOSPHORUS PENTASULPHIDE****B-1. SAMPLING**

B-1.1 General Precautions — In drawing, preparing, storing and handling samples, the following precautions shall be observed.

B-1.1.1 Samples shall not be taken in a place exposed to the weather.

B-1.1.2 The sampling instruments shall be clean and dry

B-1.1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instruments and the containers for samples from adventitious contamination.

B-1.1.4 The samples shall be placed in suitable clean, dry airtight, glass or other suitable containers on which the material has no action.

B-1.1.5 The sample containers shall be of such a size that they are almost, but not completely, filled with the sample.

B-1.1.6 Each sample container shall be sealed airtight after filling and marked with full details of sampling, the date of sampling, the year of manufacture and other important particulars of the consignment.

B-1.1.7 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

B-1.2 Scale of Sampling

B-1.2.1 Lot — All the containers in the consignment of the material drawn from a single batch of manufacture shall constitute a lot.

B-1.2.2 For ascertaining the conformity of the lot to the requirements of the specification, samples shall be tested from each lot separately

B-1.2.3 The number of containers to be sampled from each lot shall depend on the size of the lot and shall be in accordance with Table 2.

TABLE 2 SCALE OF SAMPLING

Lot Size (1)	No. of Containers to be Selected (2)
Up to 25	3
26 to 50	4
51 to 100	5
101 to 200	6
201 and above	8

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B-1.2.3.1 The sample containers shall be selected at random. In order to ensure the randomness of selection, procedure given in IS : 4905-1968* may be followed

B-1.3 Test Samples and Referee Samples

B-1.3.1 Draw with a suitable sampling instrument approximately 20 g of material from different parts of the container, divide the material so obtained in three equal parts. Each part so obtained shall constitute an individual sample representing the container and shall be transferred immediately to thoroughly dried bottles which are sealed airtight with glass stoppers. These shall be labelled with full particulars of sampling given in **B-1.1.6**. One set of test samples shall be sent to the purchaser, another to the supplier and the third kept for the referee.

B-2. NUMBER OF TESTS AND CRITERIA FOR CONFORMITY

B-2.1 Tests for all requirements shall be conducted on each of the individual samples

B-2.2 The lot shall be declared as conforming to a requirement of this specification, if all the samples tested satisfy the relevant requirement.

*Methods for random sampling

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	Unit	Symbol
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous Intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	Unit	Symbol
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	Unit	Symbol	Definition
Force	newton	N	1 N = 1 kg m/s ²
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m ²
Frequency	hertz	Hz	1 Hz = 1 c/s (s ⁻¹)
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m ²

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